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H-Induced Surface Restructuring on Si(100):

Formation of Higher Hydrides

by

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Submitted to

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Surface Science Center
Department of Chemistry
University of Pittsburgh
Pittsburgh, PA 15260

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Abstract

H-induced surface structures on Si(100) were studied using temperature programmed desorption (TPD) mass spectroscopy and low energy electron diffraction (LEED). It is shown that the (3x1) phase consists of mainly monohydride and dihydride structures, while the (1x1) phase is composed of a mixture of mono-, di-, and trihydride surface species. The trihydride surface species is associated with the phase transition between the (3x1) and (1x1) surface phases, and liberates SiH₄ and β_3 -H₂ during thermal desorption, beginning at ~ 200 K. For the fully-saturated Si(100) surface, a saturation surface coverage of 1.9 ML H has been established at a Si(100) adsorption temperature of 210 \pm 10 K. These results suggest that the 1.9 ML saturation coverage of H on Si(100) involves the presence of SiH₃(a) species, which leads to a (3x1) \rightarrow (1x1) LEED pattern change. This is contrary to a model for the (1x1) surface involving a uniform SiH₂(a) overlayer.

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I. INTRODUCTION

In this Letter, we examine the H-induced surface structures --- the (1x1) and (3x1) phases on the Si(100) surface, and show that the formation of surface trihydride species ($\text{SiH}_3(\text{a})$) during H adsorption at $T < 400$ K causes the conversion of the (3x1) phase into a (1x1) phase. The H-saturated (1x1) phase at room temperature has previously been considered as a uniform dihydride phase (two H per Si atom) with each surface Si atom relaxing to the bulk-like arrangement.¹⁻⁷ However, an ordered (3x1) phase has been recently observed by saturating the Si(100)-(2x1) surface with atomic hydrogen at a substrate temperature of 380 ± 20 K.⁸ Based on the results of low energy electron diffraction (LEED) and high-resolution infrared (IR) spectroscopy, the (1x1) phase was reassigned as a disordered phase made up of (3x1) units, where each (3x1) unit cell consists of alternating monohydride and dihydride species.⁸

Absolute coverage measurements of adsorbed hydrogen were expected to answer this dispute. Saturation coverages of 1.5 to ~2 monolayers (ML) for H/Si(100) have been reported at adsorption temperatures between 300 K and 373 K^{7,9,10} (1 ML is defined as 6.8×10^{14} H cm^{-2} , corresponding to the ideal surface Si atom density on the (100) plane.). However, simply based on the coverage measurements no detailed structural model can be provided due to lack of knowledge about the relationships between the hydrogen coverages and the populations of specific adsorbed species.

In this work, temperature programmed desorption (TPD) mass spectrometry has been used to differentiate different binding states existing on the surface, and to determine the surface coverages after H adsorption. It is shown that the saturated (3x1) phase consists of mainly monohydride (one H per Si atom) and dihydride structures, while the (1x1) phase is a surface phase with a mixture of

mono-, di-, and trihydride species. Moreover, the temperature dependence of the extent of formation of $\text{SiH}_3(\text{a})$ provides the explanation for the discrepancies between the measured saturation coverages reported by others.^{7,9,10} The saturation coverage of a (1x1) phase formed by H adsorption at 210 ± 10 K is 1.9 ML, while the saturation coverage of a (3x1) phase formed at 400 ± 10 K is ~ 1.4 ML. These results give new insights into the H-induced reconstruction on covalent surfaces, where surface reordering and disordering involving the formation of higher hydride species is found, in contrast to hydrogen behavior on metal surfaces.¹¹

II. EXPERIMENTAL

The UHV system¹² and crystal preparation¹³ involving chemical cleaning, Ar^+ bombardment, and annealing to 1200 K followed by cooling at $\sim 3 \text{ K} \cdot \text{s}^{-1}$ have been described previously. The Si(100) crystal was Czochralski grown, 10 Ω -cm, p-type(B-doped) and cut into squares of dimension 15 x 15 x 1.7 mm from an oriented ($\pm 1^\circ$) wafer. Surface cleanliness was checked by Auger electron spectroscopy (AES), and impurities after preparation were below the detectable limits of AES (< 0.01 atomic fraction within the sampling depth).¹⁴ For hydrogen dosing experiments, a W-spiral filament (diameter = 1 cm) at 1800 K was used to produce atomic hydrogen during H_2 exposure. Because of the difficulties in determining the accurate atomic hydrogen exposures, all exposures are given in terms of molecular hydrogen exposures (not corrected for ion gauge sensitivity) and reported as Langmuirs [$1 \text{ Langmuir(L)} = 1 \times 10^{-6} \text{ Torr} \cdot \text{s}$]. A 45 L H_2 exposure was used to saturate the substrate with atomic hydrogen at different adsorption temperatures, according to our coverage calibration using TPD mass spectroscopy. TPD measurements were made with a shielded, differentially-pumped

quadrupole mass spectrometer (QMS). To ensure that only species desorbing from the central area of the prepared surface were detected, the shielded QMS was equipped with a small conical axially-oriented aperture (diameter = 5mm), and during TPD experiments the crystal was accurately and reproducibly placed 2mm away from the aperture.¹² Relative surface H coverages were determined by measuring the time integral of the H₂ desorption rate above the background level throughout the desorption process. LEED patterns of the clean and H-exposed surface were measured, using a LEED apparatus equipped with digital data acquisition facilities, at a crystal temperature of 130 K. The electron beam ($< 1 \times 10^{-9}$ A, 100 V) used for LEED measurements was 1 mm in diameter. A (2x1) LEED pattern obtained from a clean Si(100) surface is shown in Fig. 1(A).

III. RESULTS AND DISCUSSION

In agreement with previous observations, different hydride phases are formed on the Si(100) surface by H adsorption at different temperatures.^{1-10,15,16} Chemisorption of atomic hydrogen at $T = 630$ K produces only the monohydride phase which exhibits a sharp (2x1) LEED pattern.^{1,4,7,15} Temperature programmed desorption from this phase yields a single β_1 -H₂ desorption state (see Fig. 2(A)). In contrast to adsorption experiments at 630 K which culminate in producing the saturated β_1 -H₂ desorption state, H chemisorption at $T = 300$ -350 K initially produces a (2x1) pattern, and according to the literature this (2x1) pattern gradually transforms into a (1x1) pattern as the coverage approaches ~ 1.5 ML.^{9,16} Major desorption features from the (1x1) phase are β_1 -H₂, β_2 -H₂, and SiH₄ desorption which are believed to correspond to the mono-, di-, and trihydride phases, respectively.¹⁶

An intermediate surface condition between the (2x1)-monohydride and the (1x1) saturated surface may be achieved by adsorption of H at 400 ± 10 K. This is

shown by the production of a (3x1) LEED pattern in Fig. 1(B). Annealing of both (1x1) and (3x1) phases to ~600 K desorbs H₂ from higher hydrides and restores a sharp (2x1) LEED pattern with all hydrogen bonded in the monohydride configuration.^{7,8,15}

We shall show next that a distinct difference between the (1x1) and (3x1) phases is the involvement of surface trihydride in the (1x1) phase; the formation of SiH₃(a) is associated with the conversion of the (3x1) long-range ordering to produce the (1x1) phase.

The H₂ TPD spectra of the (3x1) and (1x1) H-saturated phases are shown in Figs. 2(B) and 2(C), respectively. The observation of only the β_1 - and β_2 -H₂ desorption states from the saturated (3x1) phase (Fig. 2(B)) clearly indicates that this H-induced surface reconstruction includes the formation of both monohydride and dihydride phases. Experiments with the same H exposures but lower adsorption temperatures ($T < 400$ K) show a distinct decrease of the intensity of the 1/3 order LEED beams, and in the limit, a (1x1) LEED pattern is produced. Furthermore, a broad low temperature H₂ desorption feature, in addition to the β_1 and β_2 states, is observed from the (1x1) phase, as shown in Fig. 2(C). This feature, designated as the β_3 -H₂ state in this work, is produced on lowering the adsorption temperature and increasing the H coverage. The presence of the β_3 state is indicative of the formation of the (1x1) phase.¹⁷

The existence of the β_3 -H₂ desorption state during low temperature adsorption is indicative of the formation of SiH₃(a) which also produces SiH₄(g) upon thermal desorption. Figs. 3(B) and 3(C) show the TPD spectra of silane desorption from the (3x1) and (1x1) H-saturated phases, respectively. Almost no silane desorbs from the (3x1) phase. The silane desorption feature, used as an indicator of the presence of SiH₃(a) on the Si(100) surface,¹⁶ is confirmed by

comparing the mass spectrometer fragments of $m/e=28-32$, and only the major fragment at $m/e=30$ (SiH_2^+) is shown. While the (3x1) phase is nearly free of the additional adsorption states as shown in Figs. 2(B) and 3(B), the (1x1) phase clearly liberates SiH_4 and $\beta_3\text{-H}_2$ by thermal desorption as a result of the presence of surface trihydride species.¹⁸ The behavior of the SiH_4 and $\beta_3\text{-H}_2$ normalized desorption yields as a function of H adsorption temperature are shown in Fig. 4. The similar temperature dependence of the yield of the SiH_4 and $\beta_3\text{-H}_2$ desorption states suggests that the same surface species is responsible for these two desorption processes. In fact, a similar trend for the formation of $\text{SiH}_3(\text{a})$ has been observed on the $\text{Si}(111)\text{-(7x7)}$ surface by H adsorption over the temperature range of 100-400 K, using multiple internal reflection (MIR) infrared spectroscopy.¹⁹ Therefore, it is concluded that on $\text{Si}(100)$ the inhibition of $\text{SiH}_3(\text{a})$ formation during H adsorption at 400 ± 10 K maintains the (3x1) periodicity. Lowering the adsorption temperature causes the formation of $\text{SiH}_3(\text{a})$. This is associated with the conversion of the (3x1) phase into the (1x1) phase. $\text{SiH}_3(\text{a})$ reacts with additional $\text{SiH}_x(\text{a})$ ($x=1-3$) species to produce $\text{SiH}_4(\text{g})$ above ~ 200 K.

The effect of temperature on the phase transformation is also studied by annealing a hydrogenated $\text{Si}(100)$ surface. Reversible phase transitions from both (1x1) and (3x1) phases to the (2x1) phase has been confirmed by annealing to ~ 600 K.⁸ However, no transformation from the (1x1) phase to the (3x1) phase is observable by annealing a (1x1) phase to $T < 600$ K. This observation indicates that the phase transformation from the (3x1) phase to the (1x1) phase as additional hydrogen is adsorbed is an irreversible process; this observation is consistent with the view that the (1x1) phase is a disordered phase.⁸

The presence of $\text{SiH}_3(\text{a})$ on the hydrogenated $\text{Si}(100)\text{-(1x1)}$ surface leads to both SiH_4 and $\beta_3\text{-H}_2$ desorbing species. Fortunately, most of the surface

hydrogen desorbs as $H_2(g)$, and we can estimate that less than 1% of the hydrogen desorption occurs as SiH_4 in the experiments performed here by quantitatively comparing the yields of H_2 and SiH_4 in Figs. 2 and 3. Hence, the silane desorption channel is not included in our estimation of total hydrogen coverage here. The saturation coverages of hydrogen at various adsorption temperatures were determined by comparing the H_2 desorption yield to that of 1 ML hydrogen coverage which is obtained by saturating the $Si(100)-(2 \times 1)$ surface at 630 K.^{4,7,15} Saturation coverage of a (1×1) phase formed by H adsorption at 210 ± 10 K is 1.9 ML hydrogen, while the saturation coverage of a (3×1) phase formed at 400 ± 10 K is ~ 1.4 ML hydrogen (expected coverage = 1.33 ML). In addition, the ratio between the β_2 - H_2 and β_1 - H_2 desorption yields from a saturated (3×1) phase is approximately 1:3. These results support the configuration of a (3×1) phase proposed by Chabal and Raghavachari,⁸ assuming that one hydrogen atom from each surface dihydride species becomes engaged in the β_2 desorption process: $2SiH_2(a) \rightarrow H_2(g) + 2SiH(a)$.²⁰ The overall error including systematic errors of our present measurement of H coverage is $\pm 4.5\%$, based on 3-4 duplicate measurements at each coverage.

This work, in comparison to other work concerned with hydrogen coverages on $Si(100)$, is summarized in Table I, below.

A structural assignment of the (1×1) phase produced at the highest coverages at a substrate temperature of 210 K is not possible at this time. The (1×1) phase was interpreted as being a disordered phase made up of (3×1) units,⁸ and the (3×1) phase consisting of alternating $HSi-SiH(a)$ and $SiH_2(a)$ species would have a coverage of 1.33 ML H (1.5 ML H measured by Feldman et al⁹). This interpretation is inconsistent with the higher (up to 1.9 ML-H) saturation coverage measurement for the (1×1) phase shown in Table I. The involvement of $SiH_3(a)$ in disordering the (3×1) phase to produce the (1×1) phase accounts for

this higher saturation coverage. Production of $\text{SiH}_3(\text{a})$ species is indicative of Si-Si bond breaking in the $\text{Si}(100)$ surface, and subsequent $\text{SiH}_4(\text{g})$ production from $\text{SiH}_3(\text{a})$ species is consistent with Si-Si bond breaking. Our inability to determine the coverage of $\text{SiH}_3(\text{a})$ with certainty¹⁸ prevents the development of structural models for the (1×1) phase based on the population of various surface species.

IV. CONCLUSIONS

The following features of hydrogen adsorption on and desorption from $\text{Si}(100)$ have been found:

1. The sequential development of the (2×1) -monohydride, the (3×1) -monohydride + dihydride, and the (1×1) surface phase containing some trihydride species has been observed, through correlation of LEED and line-of-sight thermal desorption measurements.
2. The observation of the liberation of the $\beta_3\text{-H}_2$ from the adsorbed trihydride species is found to be closely correlated with SiH_4 liberation.
3. It has been found that the discrepancies in measured saturation H coverages reported by others are related to differences in the $\text{Si}(100)$ adsorption temperatures employed, since $\beta_3\text{-H}_2$ begins to desorb near 200 K from the (1×1) phase, and variation in the adsorption temperature in the range 200-400 K will therefore cause its saturation coverage to vary. At 210 ± 10 K a final saturation coverage of 1.9 ML H is found.
4. A structural assignment of the (1×1) surface phase, containing 1.9 ML of adsorbed H is not possible based on these studies because of uncertainties in determining the coverage of $\text{SiH}_3(\text{a})$ which is associated with $\beta_3\text{-H}_2$.

5. Since the production of $\text{SiH}_3(\text{a})$ requires Si-Si bond breaking, significant surface reconstruction may be involved in producing the (1x1) surface phase. This implies that the saturation coverage of 1.9 ML should not be rationalized on the basis of a complete and uniform layer of $\text{SiH}_2(\text{a})$ species.

V. ACKNOWLEDGEMENT

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14. Care has been taken to ensure that no carbon and metal contamination is present on the surface. The presence of a small amount of metal such as Ni on Si surfaces has been known to affect the ordering and chemistry on these surfaces. (see example, R.M. Wallace, C.C. Cheng, P.A. Taylor, W.J. Choyke, and J.T. Yates, Jr., accepted by Appl. Surf. Sci.) Also, it has been reported that the (3x1) LEED pattern cannot be obtained from H adsorption if C > 0.5% monolayer (Ref. 8).
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17. The β_3 -H₂ desorption state from the (1x1) phase was not observed previously (Ref. 16). The production of β_3 -H₂ is believed due to a lower adsorption temperature and possibly a lower heating rate during TPD experiments being used in this study. In fact, the decomposition of SiH₃(a) accompanied with the desorption of H₂ has been proposed for a hydrogenated Si(111) surface [H. Kobayashi, K. Edamoto, M. Onchi, and M. Nishijima, J. Chem. Phys. 78, 7429 (1983)], but no spectroscopic evidence for H₂ being formed from SiH₃(a) was provided. The temperature dependence of the formation of the β_3 -H₂ state on Si(100) is discussed in the text.
18. The concentration of surface trihydride species can not be determined using TPD mass spectrometry only, because thermal decomposition of SiH₃(a) into lower hydride species seems to occur during the thermal desorption process, as suggested by the β_3 -H₂ desorption (see also Ref. 17).
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20. The β_2 -H₂ desorption kinetics from the Si(100) surface are not well understood yet. However, the assumption that one H atom per SiH₂(a) species is related to the β_2 -H₂ desorption was made based on the decomposition mechanism of SiH₂(a) on porous polycrystalline silicon. (P.Gupta, V.L. Colvin, and S.M. George, Phys. Rev. B 37, 8234 (1988).)

FIGURE CAPTIONS

FIG. 1. (A) LEED pattern showing two domains of (2x1) reconstruction on a clean Si(100)-(2x1) surface. (B) LEED pattern showing two domains of (3x1) superlattice resulting from H saturation of Si(100) at 400 K. (C) A (1x1) pattern obtained by H saturation at 210 K. All data were taken at 130 K with an electron beam energy of 100 eV and with the crystal biased +90 V.

FIG. 2. TPD spectra of H₂ desorption obtained from H-saturated Si(100) at different adsorption temperatures: (a) 630 K, (b) 400 K, (c) 210 K. TPD spectra were taken with a heating rate of 1.7 K/s after the crystal was cooled down to 130 K.

FIG. 3. TPD spectra of SiH₄ desorption corresponding to the experimental conditions described in Fig. 2.

FIG. 4. Behavior of SiH₄ and β_3 -H₂ desorption yields as a function of substrate temperature during H adsorption. All yields are normalized to that obtained at $T_{ads} = 210$ K.

Table I. Comparison of H-saturation coverages measured at different adsorption temperatures and for different surface phases - Si(100).

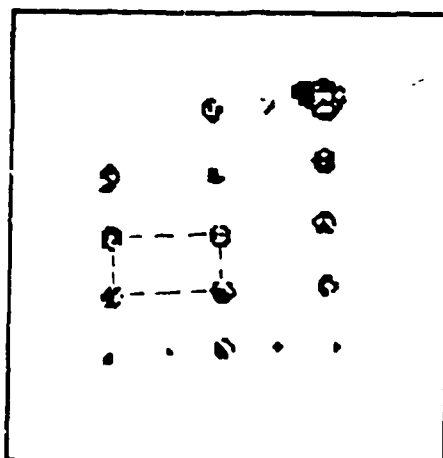
H-saturation coverage (ML)			Experimental method used	References
(2x1)	(3x1)	(1x1)		
1.0 (630 K)	1.4 (400 K)	1.9 (210 K)	line-of-sight TPD	this work
1.0 (670 K)	1.7 (373 K)	1.85 (300 K)	elastic recoil detection analysis	a
--	--	1.5 and 1.7 (<400 K)*	nuclear reaction analysis	b and c

*The measurement made in this work involves D/Si(100) due to the nature of the method used. Substrate temperatures during H adsorption were not emphasized in these two works, but based on the observed (1x1) LEED pattern a substrate temperature of less than ~400 K is assumed.

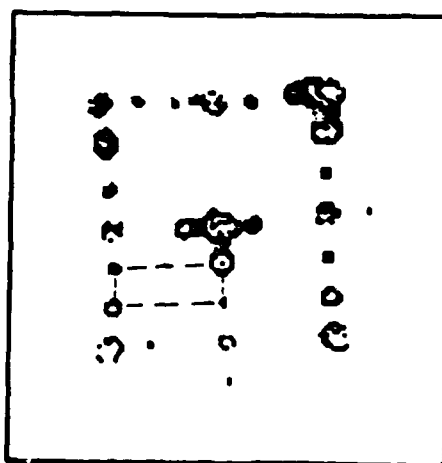
- a. Ref. 7
- b. Ref. 9
- c. Ref. 10

LEED Pattern Development for Increasing Hydrogen Coverage on Si(100)-(2x1)

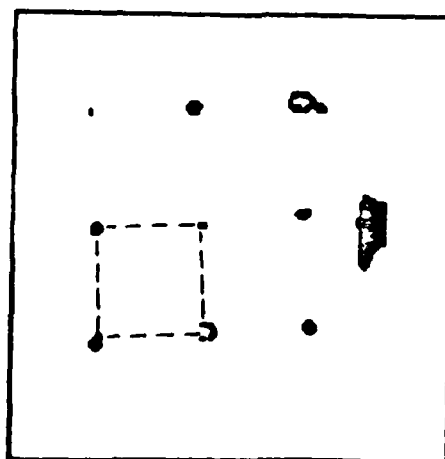
(A) clean Si(100)-(2x1)



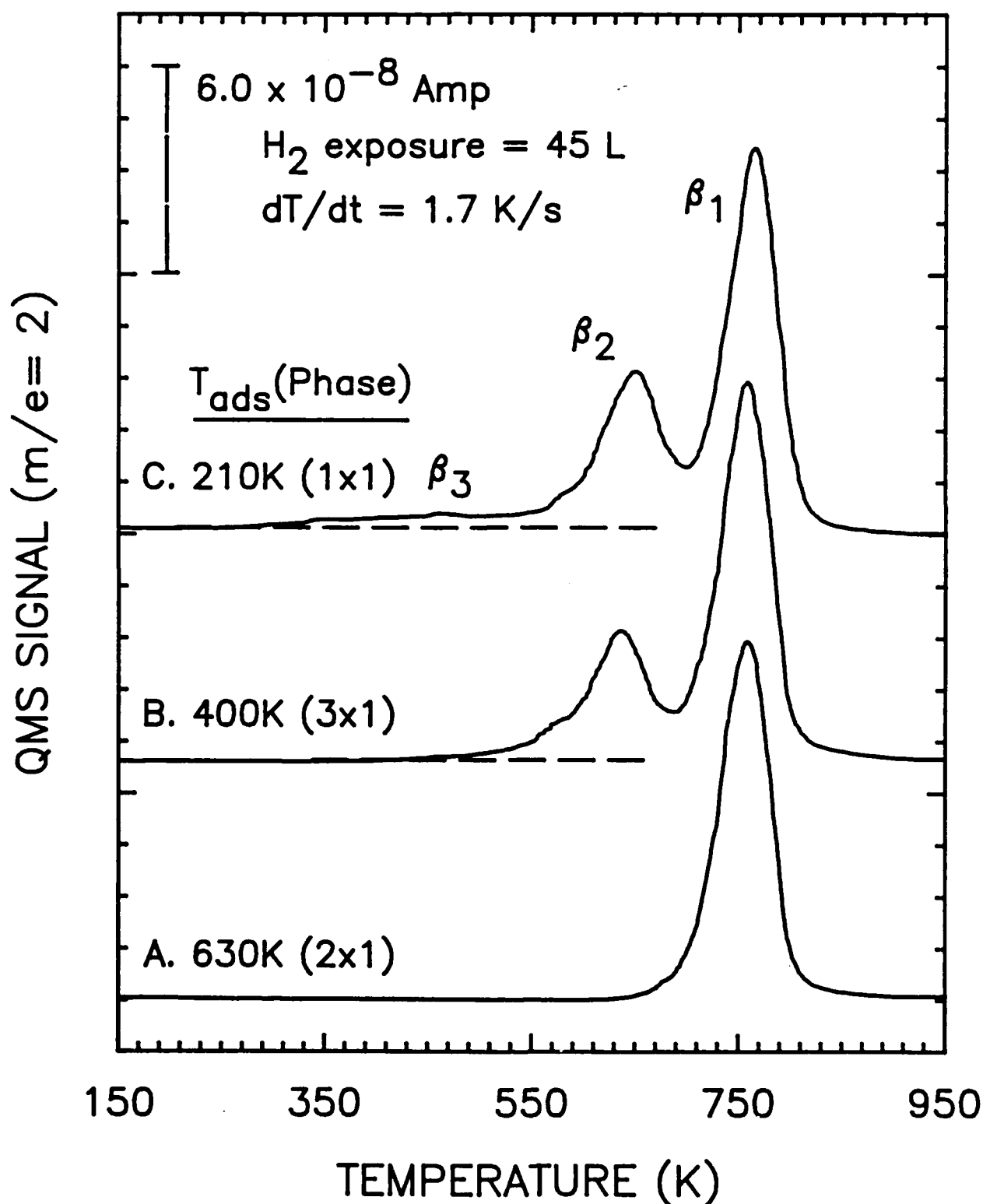
(B) H-saturated (3x1) phase



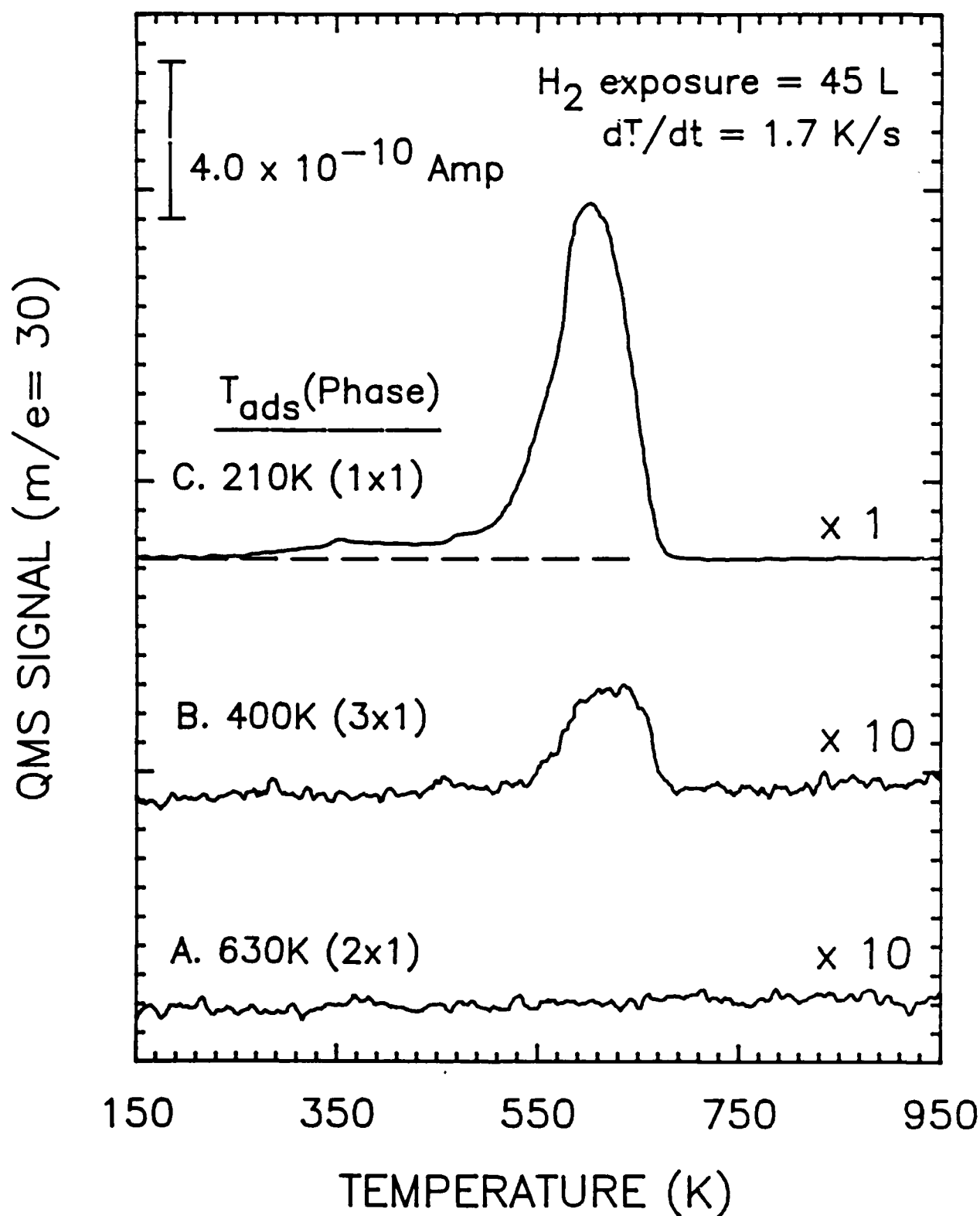
(C) H-saturated (1x1) phase



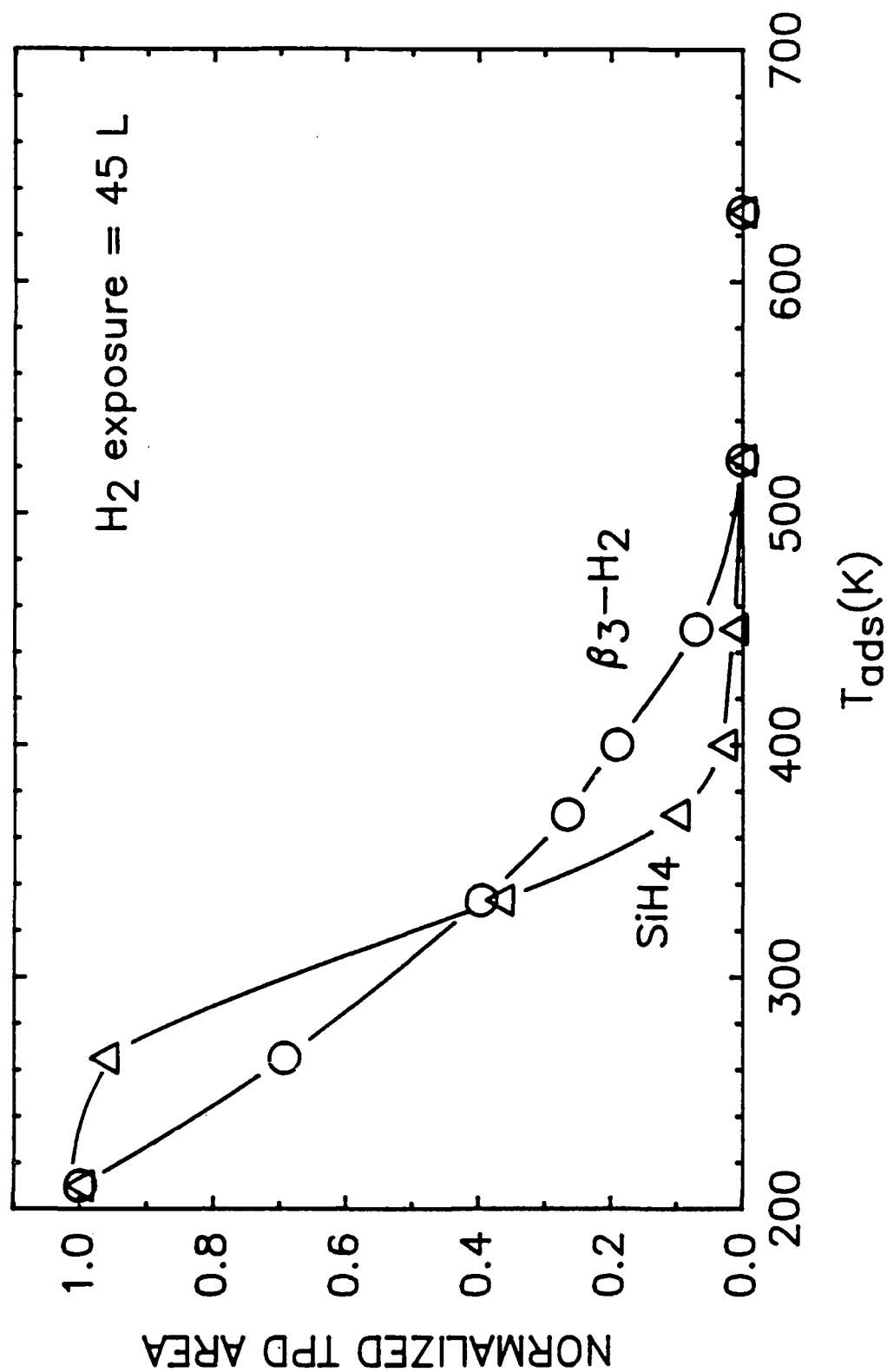
TPD SPECTRA OF H₂ DESORPTION FROM H/Si(100) WITH DIFFERENT ADSORPTION TEMPERATURES



TPD SPECTRA OF SiH_4 DESORPTION FROM $\text{H}/\text{Si}(100)$ WITH DIFFERENT ADSORPTION TEMPERATURES



BEHAVIOR OF SiH_4 AND $\beta_3\text{-H}_2$ DESORPTION YIELDS VS.
SUBSTRATE TEMPERATURE DURING H ADSORPTION



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